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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>7</sup> :</b> <b>C10G 1/04</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 00/22067</b> <b>(43) International Publication Date:</b> 20 April 2000 (20.04.00)
<b>(21) International Application Number:</b> PCT/CA99/00784 <b>(22) International Filing Date:</b> 31 August 1999 (31.08.99)  <b>(30) Priority Data:</b> 2,249,110 13 October 1998 (13.10.98) CA 2,276,944 13 July 1999 (13.07.99) CA  <b>(71) Applicant (for all designated States except US):</b> MAUVIN MATERIAL AND CHEMICAL PROCESSING LTD. [CA/CA]; RR #7, Perth, Ontario K7H 3C9 (CA).  <b>(71)(72) Applicant and Inventor:</b> DI TULLIO, Venanzio [CA/CA]; RR #7, Perth, Ontario K7H 3C9 (CA).  <b>(72) Inventor; and</b> <b>(75) Inventor/Applicant (for US only):</b> SEALES, Gary [US/US]; 4620 Briar Hill Road, Cortland, NY 13045 (US).		<b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i> <i>With amended claims and statement.</i>
<b>(54) Title:</b> SEPARATION OF TAR, OILS AND INORGANIC CONSTITUENTS FROM OIL BEARING SANDS OR SHALES  <b>(57) Abstract</b>  An improved Process for the total separation and recovery of four constituents, namely 1) insoluble pitches and tars also known as asphaltenes, 2) a kerosene based oil fraction, 3) clays and silts of less than 80 $\mu$ m mesh and 4) sands of greater than 80 $\mu$ m mesh. Recombination of the hydrocarbon fractions is the bitumen portion of Tar Sands. A further process for the extraction and separation of plant resins from cellulose and kerogen from oil shale that on thermal depolymerization become a source for aromatic and kerosene based oil fractions respectively.		

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**TITLE**

SEPARATION OF TARS, OILS AND INORGANIC CONSTITUENTS FROM OIL BEARINGS SANDS OR SHALES

**Specifications / Background of the Invention**

This invention relates to the separation and isolation of oil sand aggregates into four components: 1) tars and pitches, 2) a kerosene fraction, 3) clays, silts (particle sizes of less than 80  $\mu\text{m}$ ) and 4) sand (particles sizes of greater than 80 $\mu\text{m}$ ).

It is known that oil sands can be separated and oil fractions isolated by one of many processes of which the ranked highest to lowest preference is the:

- a) CHWE (Clark Hot Water Extraction Process) [1],
- b) OSLO HWE (Oslo Hot Water Extraction Process) [2],
- c) OSLO CWE (Oslo Cold Water Extraction Process) [2],
- d) AOSTRA – Takiuk Process [3],
- e) ZEFTE (Zero Fine Tailings Extraction Process) [4], and
- f) BITMIN (Counter Current Desander Process) [5].

[1] FTFC (Fine Tailings Fundamentals Consortium) "Vol 4 –29. Laboratory Experiments on the Clark Process" In: *Advances in Oil Sands Tailings Research*, Alberta Department of Energy, Oil Sands and Research Division, Publisher.

[2] FTFC (Fine Tailings Fundamentals Consortium) "Vol 4 –9. OSLO Hot and Cold Water Extraction Processes" In: *Advances in Oil Sands Tailings Research*, Alberta Department of Energy, Oil Sands and Research Division, Publisher.

[3] FTFC (Fine Tailings Fundamentals Consortium) "Vol 4 –6. AOSTRA – Takiuk Process" In: *Advances in Oil Sands Tailings Research*, Alberta Department of Energy, Oil Sands and Research Division, Publisher.

[4] FTFC (Fine Tailings Fundamentals Consortium) "Vol 4 –8. Z ro Fine Tailings Extraction (ZEFTE)" In: *Advances in Oil Sands Tailings Research*, Alberta Department of Energy, Oil Sands and Research Division, Publisher.

[5] FTFC (Fine Tailings Fundamentals Consortium) "Vol 4 –8. BITMIN" In: *Advances in Oil Sands Tailings Research*, Alberta Department of Energy, Oil Sands and Research Division, Publisher.

This invention relates to the separation and isolation of resins and kerogens.

It is also known that resinous and wax like products can be separated from their host habitat by means of prolonged continuous Soxhlet extraction. The efficacy of extraction has been substantially increased by expanding the range of solvents used in analytical extraction tools such as Solid Phase Extraction (SPE) [6], Supercritical Fluid Extraction (SFE) [7], Pressure Fluid Extraction (PFE) [8], Accelerated Solvent Extraction (ASE) and Microwave – Accelerated Solvent Extraction (M-ASE) [9].

[6] Zief, M., Kieser, R., *Solid Phase Extraction for Sample Preparation*. Mallinckrodt Baker Inc. 1997.

[7] R.E Majors LC / GC 17(6s) 8 – 13 (1999)

[8] Richter, B.E. LC / GC 17(6s) 22 – 28 (1999)

[9] Le Blanc, G., LC / GC 17(6s) 32 -36 (1999)

To date, applications have involved micro analytical extraction of organic analytes from solid phases. One commercial application (known as the ALCELL PROCESS) involves the extraction of lignin from wood. A solvent mixture of methanol, ethanol and water at a pressure of 35 atmospheres is used to extract lignin from wood fiber[10].

[10] Lora, J.H. et al. US Pat. 5865948

This invention, using thermal heating (preferably microwave), applies the micro analytical benefits of Accelerated Solvent Extraction to commercial applications. It extends the efficacy of the process of reduced extraction time, reduced solvent consumption and increased extraction efficiencies by introducing a temperature controllable biphasic solvent system i.e. a system that is the result of a mixture of an organic solvent and water which exhibits a Lower Critical Solution Temperature (LCST).

SOME OF THE INHERENT PROBLEMS ASSOCIATED WITH SOME OR ALL OF THE ABOVE PROCESSES (a – f) ARE:

Energy Usage:

The processes require large net input of thermal and / or mechanical energy.[11]

[11 Strand, W. L.; Canadian Pat. 2 124 199 (1992 06 11)]

Tailings and Storage Space:

They also generate large quantities of tailings and require indefinite storage space.  
[11]

Bitumen Yields:

Except for the AOSTRA-T Process, unacceptably low yields (54 – 92%) of bitumen are separable from the tar sands using present day technology. In fact, yields of 92 – 96% are considered to be high using the present art. [12]

[12] Sparks B.D., Majid A., Woods J.; Canadian Pat. 2 093 142 (1994 09 27)

In this invention yields of 99% are considered low from any and all of the ore bodies found in Alberta, Canada, the San Joaquin Valley of California and along the shores of the Orinoco River in Venezuela.

Hence, not only can more oil be squeezed out of less ore but utilization of the steps in our invention makes access to the lower grade ores economically viable.

Water usage:

Again, except for the AOSTRA-T Process in a – f above, large volumes of water are used in the extraction of bitumen. On average 0.7 to 3 MT of water are required per Metric Ton of ore (depending on the bitumen content of the ore). The lower the bitumen content the higher the volume of water required. Presently, in the case of the 12% bitumen content ore, 420,000 MT of water are required per day of full operation.[13]

[13] FTFC (Fine Tailings Fundamentals Consortium) "Vol. 2 – 3 " In: *Advances in Oil Sands Tailings Research*, Alberta Department of Energy, Oil Sands and Research Division, Publisher.

Environmental Concerns:

Because the spent water presently generated contains toxic naphthenates, oil residues, and fine tailings, storage and containment of the waste waters has become an integral part of the process. The presently projected required volume of settling ponds doubles every 400 days. This is expected to decrease to 300 days when the Aurora mine comes on stream in the year 2004 i.e. 460,000,000 m<sup>3</sup> per annum of new storage space for spent water shall be required.

It has been estimated that it will take 100 – 300 years for the colloidal of the fine tailings to agglomerate to a soft clay before release of the above mentioned waters shall be permitted to the environment. " *Without further treatment of the existing fine tailings and without process modifications to reduce the rate of production of "new" fine tailings, by the year 2030, over one billion cubic meters of a non-consolidating fine tailings would exist at the bottom of these lakes.* " ...since "Containment of the entire water system with the operating process is required as part of the operating license agreement between the Provincial Government and the two commercial plants." [14, 15]

[14] FTFC (Fine Tailings Fundamentals Consortium) "Vol. 4 -5. " In: *Advances in Oil Sands Tailings Research*, Alberta Department of Energy, Oil Sands and Research Division, Publisher.

[15] Mac Kinnon, M. and Sethi, A.; *A Comparison of the Physical and Chemical Properties of the Tailings Ponds at the Syncrude and Suncor Oil Sands Plants, Oil Sands\_ Our Petroleum future Conference, Edmonton, Alberta, April 4-7, 1993.*

#### AOSTRA Taciuk Process [16]

An advantage of the AT Process is that no toxic tailings are generated. Extra energy costs incurred by the process are partially offset by elimination of treatment and maintenance costs of the wastewater containment ponds. Although the process is self sufficient, the expended energy and specialty equipment must be costed against the process. Our process minimizes such cost while providing the opportunity to sell the energy to the open market.

[16] FTFC (Fine Tailings Fundamentals Consortium) "Vol. 4 -10. " In: *Advances in Oil Sands Tailings Research*, Alberta Department of Energy, Oil Sands and Research Division, Publisher.

#### Solid Phase Extraction Processes

Solid Phase Pressure Extractions have, to date, been limited to micro analytical applications. The ALCELL PROCESS has shown that high pressures can have economic restrictive effects on commercial applications.

#### **SUMMARY OF THE INVENTION**

The present invention provides a process whereby trapped and bound bitumen can be removed from an inorganic agglomerate of various size particles. Upon detachment and because of the ability of the solvent to physically set up a phase mixture system which has inherent density and solubility extremes, tars can be separated from oils and sand or diatomaceous earths can be separated from clays and silts.

Such solvent mixtures have the ability to separate into biphasic mixtures simply by adjusting the temperature of the solution or by changing its inorganic salt concentration.

The separating solvent solution is an aqueous mixture of lipophilic liquids that exhibit a Lower Critical Solution Temperature.

Some liquids exhibit total solubility over a range of concentrations and temperatures but partition into biphasic systems at specific concentrations and temperatures.

They possess the specific ability to raise the lipophilic and hydrophilic characteristics of a solution by simple manipulation of the process variables. In other words, simple adjustment of the salt concentration or temperature greatly expands the separation abilities of the constituent solvents.

An example is Butoxy Ethanol in water. Solutions of greater than 10% and less than 57% Butoxy Ethanol will, below approximately 40°C remain in solution but partition into a biphasic system above 40°C.

For example, 100 ml of totally miscible Butoxy Ethanol (density 0.90 g/ml) will, at 50°C give a biphasic system of 10 mls 57% Butoxy Ethanol in Water as a top phase (density 0.92 g/ml) and 90 mls of 10% Butoxy Ethanol in Water as a bottom phase (density 0.99 g/ml).

Such phenomena are known as Lower Critical Solution Temperatures. When the reverse phenomena is exhibited i.e. a biphasic mixture at a low temperature becomes a single phase at a higher temperature the solvents are said to have an Upper Critical Solution Temperature (UCST). Some mixtures do not exhibit an UCST at atmospheric pressure only because their boiling points are lower than their UCST's. In order to exhibit an UCST it becomes imperative that the solvent solution be held under pressure while being heated.

The present invention provides a method of separating the organic from the inorganic phase in tar sands with a recyclable liquid composition whose LCST is above 40°C comprising:

Sodium silicate .....	0 – 2.5%
Sodium hydroxide .....	0 – 2.5%
Alkyl or di alkyl glycol or di glycol ether and / or	
Propyl glycol ether .....	Ingredient dependant
Triethyl amine and / or diethyl methyl and / or dimethyl pyridine and / or	

*SUBSTITUTE SHEET (RULE 26)*



methyl pyridyl and / or methyl piperidene .....0 – 10%

Water .....to 100%

Pressure ... 1 – 3 atmospheres depending on the  $T_g$  of the tars being extracted.

In preferred embodiments of the inventions the following proportions of components can be used.

Sodium Hydroxide and/or Sodium Silicate 0-2.5%, preferably 0.5 to 2.5, particularly preferable 1-2%

All glycol ethers 0-100%, preferably 10 to 60 particularly 15 – 25%, especially 20%.

#### ADVANTAGE OF OUR PROCESS OVER THE PAST ART

Some obvious advantages of the process are:

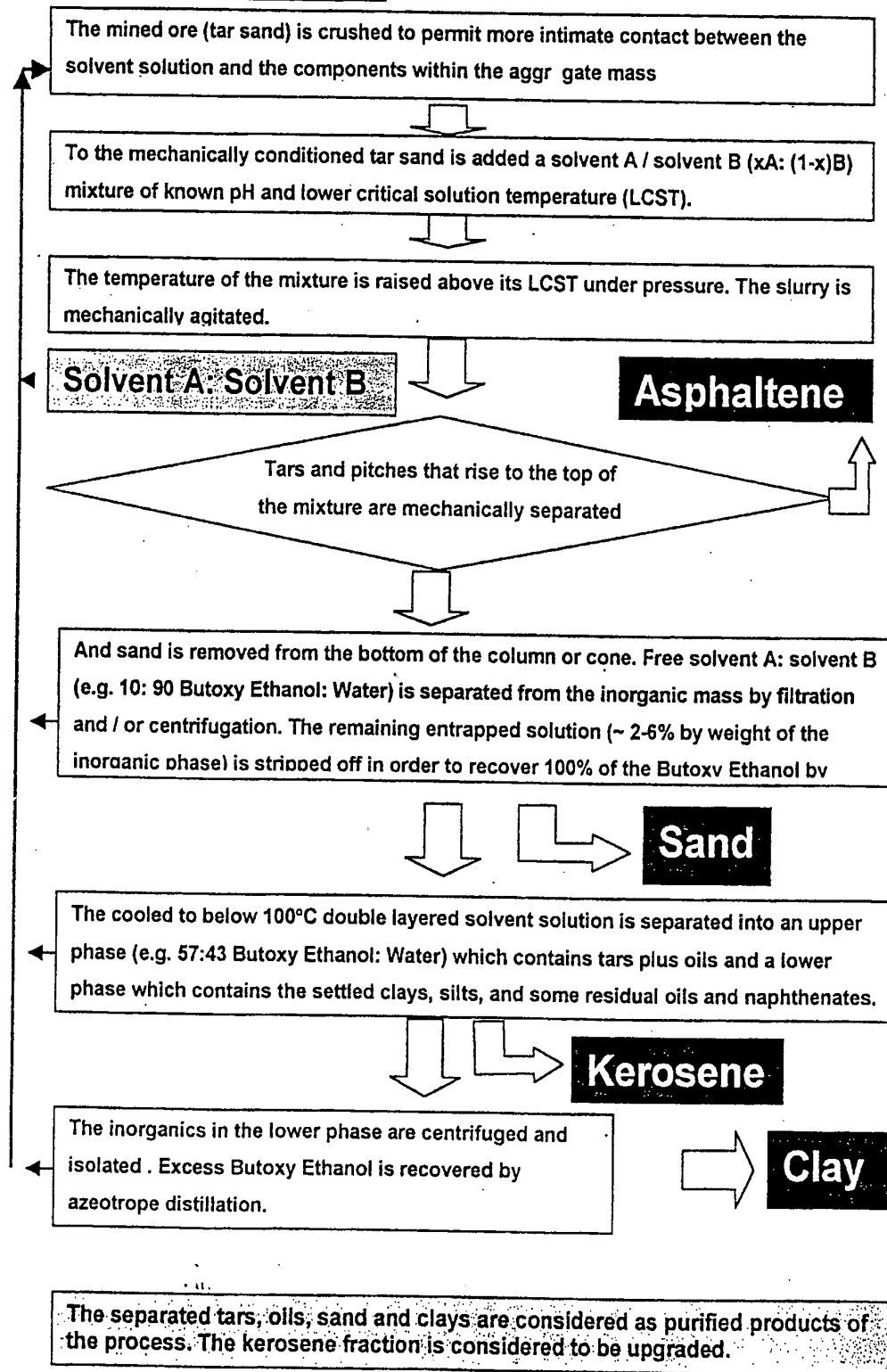
1) Simplicity of the equipment and the process	Reduction in capital costs and maintenance fees.
2) Raising the recovery rates of the hydrocarbon fraction well above 92%	Between 15 and 30,000 extra barrels per day over the Clark Process.
3) Making the raw material source usage of the low grade Alberta ore (6- 10% Bitumen content) economically viable	Some where between 90 and 120 B\$US.
4) Facile separation of the solid hydrocarbons from their liquid counterparts	Short cycle time and therefore reduced equipment size requirements.
1) Making the raw material source usage of the tar sands from California and Venezuela economically viable	Capability to extract oil from non-connated tar sands
6) Concentration of the liquid hydrocarbon fraction by partitioning it between the two phases of the solvent mixture at temperatures just above the LCST	Lowering the process temperature to just above 40°C to remove oils and above 100°C to remove heavy tars.
7) Generate two separate hydrocarbon streams at the mine site (Asphaltenes and liquid hydrocarbons)	Eliminate transportation of the inorganic phases.

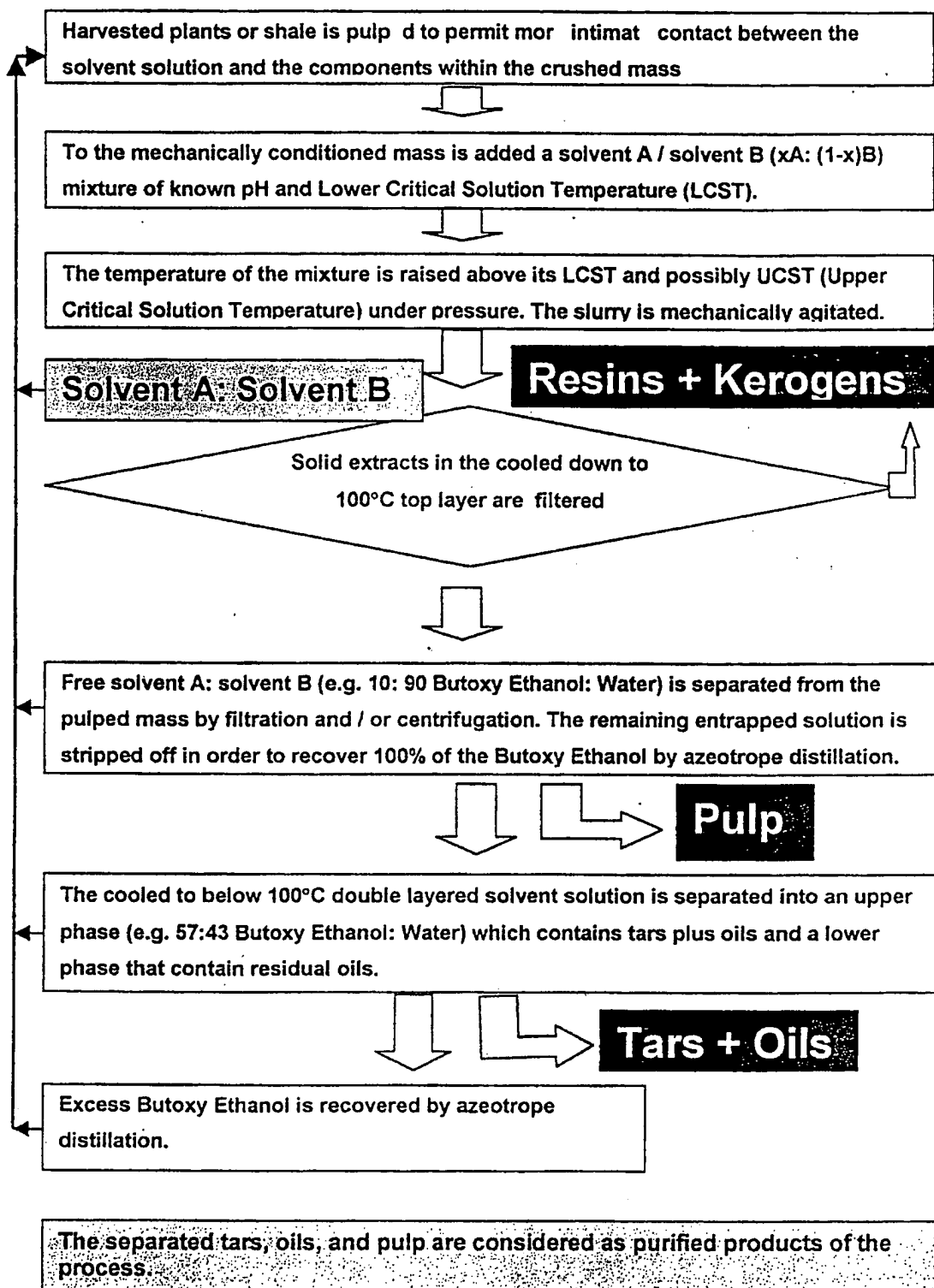
8) Generate a solvent system which has a Freezing point of $-10^{\circ}\text{C}$	This is more of a pour point than a freezing point.
9) Work with non – flammable solvents i.e. flash points above $100^{\circ}\text{C}$	Insurance premiums should be low.
10) Reduce energy usage	Process ores just above $40^{\circ}\text{C}$
11) Eliminate the need for waste water Containment ponds	No toxic waste and no fine tailings.
12) Eliminate the projected volumes of toxic fine tails	Holding ponds not needed.
13) Eliminate the need for Tailings Oil Recovery	No wastewater.
14) Recycle the solvent system in a closed cycle	Recovery of the naphthenates and thio compounds.
15) Segregation of the mineral clays from the sand for further processing	Recovery of >99% pure silicon dioxide
16) Provide the opportunity to recover precious metals from the inorganic isolants.	
17) Extension of the process to cleaning up man made spills	
18) Reduction of the extraction process to 3 – 4 minutes using a solvent system under pressure	Decreasing the cycle time and reducing the extraction process to in (pipe) line processing.
19) Reduction of the solvent volumes to half the weight of the sand being processed.	

#### Experimental Example

- 1) To a 6-12% by weight sample of tar sand add an equivalent weight of greater than 10% by volume Butoxy Ethanol in Water. The solvent mixture may contain up to 0.75% of sodium hydroxide and *meta* sodium silicate respectively.
- 2) The mixture is stirred and a stream of air introduced while being heated above  $40^{\circ}\text{C}$ .
- 3) Heating the mixture above  $40^{\circ}\text{C}$  causes the liquid to separate into two layers or phases. The upper layer and lower layers are 57:43 and 10: 90 solutions of Butoxy Ethanol: Water respectively.
- 4) Tars and pitches (Asphaltenes) whose densities are less than 0.99 g/cc rise to the upper layer. Those which are greater than 0.92 and less than 0.99 g/cc rise to the interface between the two layers.

- 5) The asphaltenes can now be isolated by filtering / centrifuging, those which are suspended in the liquid, and by skimming those surfaces on which they have been deposited.
- 6) The asphaltenes are further processed at the refinery level.
- 7) The sand found at the bottom of the column or cone is further washed with an equivalent weight of fresh BE: Water at a temperature of 120 to 130 °C to ensure all tar has been removed. Bitumen free sand is passed through a centrifugal thickener as is used in the paper industry. The semi dry, silt free sand is flashed in order to azeotropically recover all butoxy ethanol. The purified sand (greater than 99% SiO<sub>2</sub>) can be used as an abrasive or by the glass industry. Coarser sands found in the San Joaquin samples can be sieved for construction industry use.
- 8) The clay collects on top of the sand. Agitation causes the fine particles to separate from the larger sand particles.
- 9) Within the scope of our experiment we used an aspirator attached to a pasteur pipette to collect the clay. Heating the clay in the presence of the 120-130°C solvent ensure bitumen free clay is formed. The clay is azeotropically dried.
- 10) Separation of the clay and bitumen is attained by centrifugation.
- 11) Depending on the source of the ore, the cleaned clays (mainly kaolinite and illite) may have commercial applications or precious metal extraction possibilities.
- 12) The kerosene fraction is found dissolved in the top layer. It is recovered by fractional distillation.
- 13) All recovered solvents and washings are recycled. They can be used "as is" in a primary extraction step or after purification by distillation.
- 14) Bitumen yields of greater than 99% are attainable.

**Flow Chart of the Tar Sands Process:**

Flow Chart of the Terrestrial Plant and Shale Processes:

**CLAIMS**

The embodiment of the invention in which an exclusive property or privilege is claimed and defined as follows:

- 1) A process which causes the separation of interstitial bitumen that is entrapped and bound to a substrate such as sand and mineral clays in oil sands and shales.
- 2) A process as described in claim 1, which causes the separation of bitumen that is strongly bound to a substrate such as mineral clays in tar sands.
- 3) A cleansing action consisting of treating a substrate surface and / or its interstitial areas with a "Solvent xA: Solvent (1-x)B" mixture which has a Lower Critical Solution Temperature (LCST).
- 4) A cleansing action consisting of treating a substrate surface and / or its interstitial areas with a "Solvent xA: Solvent (1-x)B" mixture under pressure above its boiling point.
- 5) A process as defined in claim 2 in which the solvent has a pH range from less than 1 to more than 14 but preferably between 7 and 12.
- 6) A process as defined in claim 2, in which the solvent mixture is controlled by the addition of either inorganic or organic acids and bases.
- 7) A process as defined in claim 2 in which the ionic strength and hence the LCST is controlled by the addition of salts.
- 8) A process as defined in claim 2 in which the freezing point is controlled by the ratios of the solvents in the mixture.
- 9) A process as defined in claim 2 in which injecting the solution in a counter current manner and at a temperature less than the LCST and to heat the solution as it moves through the ore body.

- 10) A process as defined in claim 9 in which the lower temperature solvent starts to remove the oils from the ore and the higher temperature solvent phase concentrates the oils.**
- 11) A process as defined in claim 9 in which a higher temperature solvent removes viscous tars from the surface of the mineral ore.**
- 12) A process as defined in claim 2 in which the ore moving in a downward direction and the liquid in reverse direction (i.e. counter current) so that introduction of air within the ore mass further urges the lower density asphaltenes, to rise towards the top of the mixing chamber.**
- 13) A process as defined in claim 2 in which the ore moving in a downward direction and the liquid in reverse direction (i.e. counter current) so that introduction of air within the ore mass further urges the smaller sized particles to rise towards the top of the mixing chamber.**
- 14) A process as defined in claim 2 in which the ore moving in a downward direction and the liquid in reverse direction (i.e. counter current) so that introduction of air within the ore mass further urges the upper phase liquid to rise towards the top of the mixing chamber.**
- 15) A process as defined in claim 2 in which the solid ore gradually distributes itself into "sized particle layers" of clay silt and sand that can be shunted to further refining processes.**
- 16) A process as defined in claim 2 in which the column of liquid is comprised of an upper layer of lower density that contains the major portion of the oils and a lower layer of higher density that contains the extracted inorganic salts, naphthenates and thio compounds.**
- 17) A process as defined in claims 12, 13 and 14 in which both isolated layers can be removed for further processing.**

- 18) A process as defined in claim 12, 13, and 14 in which either liquid phase can be r generated by azeotropic distillation.**
- 19) A process as defined in claim 15 in which the solvent and solids in the lower layer or phase can be mechanically claimed by a Double Nip Thickener (DNT) or centrifuge and the sand or clays dried by azeotropic distillation of the remaining solvents.**
- 20) A process as defined in claim 2, which can be executed at a lower temperature and hence require less energy.**
- 21) A process as defined in claim 2, which can be executed with non-flammable solvent mixtures.**
- 22) A process as defined in claim 2 which can be used to hydrostatically transport solid ores at less than 0°C temperatures.**
- 23) A process as defined in claim 2 which eliminates toxic wastewater.**
- 24) A process as defined in claim 2 which eliminates the need for holding ponds.**
- 25) A process which provides a mechanism to separate the oils from mineral ores much closer to the mine site because of the reduced size of the required processing equipment.**
- 26) A process which, on a small scale, can be mounted on a transportation vehicle and which, in the case of oil spillage and / or contamination of the land could be used to rejuvenate said land. An example would be sand on beaches that have become oil laden because of oil spillage disasters on the high seas. Another example would be contamination of a land site as the result of spillage and / or leakage of oils from holding tanks.**



**AMENDED CLAIMS**

[received by the International Bureau on 29 February 2000 (29.02.00);  
original claims 2 and 24-26 cancelled; original claims 1, 3-5, 10-11 and 17-19 replaced by amended  
claims 1-4, 9-10 and 16-18; remaining claims unchanged but renumbered accordingly (3 pages)].

The embodiment of the invention in which an exclusive property or privilege is claimed and defined as follows:

- 1) A process which causes the separation of bitumen from a substrate such as sand and mineral clays in oil bearing sands and shales.
- 2) A cleansing action consisting of treating a substrate surface and / or its interstitial areas with a "Solvent A (EG water) : Solvent B (EG Butoxy Ethanol) " mixture that exhibits a Lower Critical Solution Temperature (LCST).
- 3) A cleansing action consisting of treating an oil sand with a "Solvent A: Solvent B" mixture above 100°C and more specifically 120-130°C.
- 4) A process as defined in claim 2 in which the solvent has basic salts such as sodium hydroxide and sodium meta silicate present.

- 5) A process as defined in claim 2, in which the solvent mixture is controlled by the addition of either inorganic or organic acids and bases.**
- 6) A process as defined in claim 2 in which the ionic strength and hence the LCST is controlled by the addition of salts.**
- 7) A process as defined in claim 2 in which the freezing point is controlled by the ratios of the solvents in the mixture.**
- 8) A process as defined in claim 2 in which injecting the solution in a counter current manner and at a temperature less than the LCST and to heat the solution as it moves through the ore body.**
- 9) A process as defined in claim 8 in which the lower temperature solvent starts to remove the oils from the ore and the higher temperature solvent phase concentrates the oils.**
- 10) A process as defined in claim 8 in which a higher temperature solvent removes viscous tars from the surface of the mineral ore.**
- 11) A process as defined in claim 2 in which the ore moving in a downward direction and the liquid in reverse direction (i.e. counter current) so that introduction of air within the ore mass further urges the lower density asphaltenes, to rise towards the top of the mixing chamber.**
- 12) A process as defined in claim 2 in which the ore moving in a downward direction and the liquid in reverse direction (i.e. counter current) so that introduction of air within the ore mass further urges the smaller sized particles to rise towards the top of the mixing chamber.**
- 13) A process as defined in claim 2 in which the ore moving in a downward direction and the liquid in reverse direction (i.e. counter current) so that introduction of air within the ore mass further urges the upper phase liquid to rise towards the top of the mixing chamber.**

- 14) A process as defined in claim 2 in which the solid ore gradually distributes itself into "sized particle layers" of clay silt and sand that can be shunted to further refining processes.
- 15) A process as defined in claim 2 in which the column of liquid is comprised of an upper layer of lower density that contains the major portion of the oils and a lower layer of higher density that contains the extracted inorganic salts, naphthenates and thio compounds.
- 16) A process as defined in claims 11, 12 and 13 in which both isolated layers can be removed for further processing.
- 17) A process as defined in claim 11, 12, and 13 in which either liquid phase can be regenerated by azeotropic distillation.
- 18) A process as defined in claim 14 in which the solvent and solids in the lower layer or phase can be mechanically claimed by a Double Nip Thickener (DNT) or centrifuge and the sand or clays dried by azeotropic distillation of the remaining solvents.
- 19) A process as defined in claim 2, which can be executed at a lower temperature and hence require less energy.
- 20) A process as defined in claim 2, which can be executed with non-flammable solvent mixtures.
- 21) A process as defined in claim 2 which can be used to hydrostatically transport solid ores at less than 0°C temperatures.
- 22) A process as defined in claim 2 which eliminates toxic wastewater.

**STATEMENT UNDER ARTICLE 19****Amendment explanations:**

<b>Old claim version</b>	<b>New claim version</b>	<b>Explanation</b>
<b>Claim 1</b>	<b>Claim 1</b>	<b>Claim 1 clarified</b>
<b>Claim 2</b>	<b>Cancelled</b>	<b>Cancelled</b>
<b>Claims 3-5</b>	<b>Claims 2-4</b>	<b>Adds clarity and are directly supported by disclosures on pages 8-9 of the application. Claims are consecutively re-numbered.</b>
<b>Claim 6-9</b>	<b>Claims 5-8</b>	<b>Claims are consecutively re-numbered.</b>
<b>Claims 10-12</b>	<b>Claims 9-11</b>	<b>Claims amended to fit the new numbering structure.</b>
<b>Claims 13-16</b>	<b>Claims 12-15</b>	<b>Claims are consecutively re-numbered.</b>
<b>Claims 17-19</b>	<b>Claims 16-18</b>	<b>Claims amended to fit the new numbering structure.</b>
<b>Claims 20-23</b>	<b>Claims 19-22</b>	<b>Claims are consecutively re-numbered.</b>
<b>Claims 24-26</b>	<b>Cancelled</b>	<b>Cancelled</b>

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/CA 99/00784

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7 C10G1/04

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C10G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DESNOYERS ET AL: "tar sand extractions with microemulsions: I the dissolution of light hydrocarbons by microemulsions using 2-butoxyethanol and diethylamine as cosurfactants." THE CANADIAN JOURNAL OF CHEMICAL ENGINEERING, vol. 61, October 1983 (1983-10), pages 672-679, XP002125486	
A	GB 733 892 A (COULSEN) 20 July 1955 (1955-07-20)	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

## \* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

14 December 1999

Date of mailing of the international search report

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
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Authorized officer

De Herdt, O

# INTERNATIONAL SEARCH REPORT

I. national application No.  
PCT/CA 99/00784

## Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:
  
2. ☒ Claims Nos.: 1 2 3 4 5-24 25 26  
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:  
see FURTHER INFORMATION sheet PCT/ISA/210
  
3. ☐ Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box II Observations where unity of invention is lacking (Continuation of Item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
  
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
  
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
  
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

## FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

Claims Nos.: 1 2 3 4 5-24 25 26

Present claims 1,2,3,4,5-24, 25 and 26 relate to a method defined by reference to a desirable characteristic or property, namely the separation of bitumen from oil sand and shales.

The claims cover all methods having this characteristic or property, whereas the application provides support within the meaning of Article 6 PCT and/or disclosure within the meaning of Article 5 PCT for only a very limited number of such methods. In the present case, the claims so lack support, and the application so lacks disclosure, that a meaningful search over the whole of the claimed scope is impossible. Independent of the above reasoning, the claims also lack clarity (Article 6 PCT). An attempt is made to define the method by reference to a result to be achieved. Again, this lack of clarity in the present case is such as to render a meaningful search over the whole of the claimed scope impossible. Consequently, the search has been carried out for those parts of the claims which appear to be clear, supported and disclosed, namely those parts relating to the method mentioned in the example at pages 8 - 9.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/CA 99/00784

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB 733892	A	NONE	